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टार और बिटुमन सामग्रियों के लिए परीक्षण  
पद्धतियाँ — बिटुमेन सामग्री का नमूनीकरण  
( दूसरा पुनरीक्षण )

Methods for Testing Tar and  
Bituminous Materials — Sampling of  
Bituminous Materials  
( Second Revision )

ICS 75.140

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## FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Bitumen, Tar and Related Products Sectional Committee, had been approved by the Petroleum, Coal and Related Product Division Council.

This standard was originally published in 1958 as 'Methods for testing tar and bituminous materials — Sampling' and first revised in 1978. 'Methods for testing tar and bituminous materials' was originally published as series of 22 standards in the form of a booklet, as listed below:

SI No.	IS Designation	Title
1.	IS 1201 : 2004	Sampling
2.	IS 1202 : 1978	Determination of specific gravity
3.	IS 1203 : 1978	Determination of penetration
4.	IS 1204 : 1978	Determination of residue of specified penetration
5.	IS 1205 : 1978	Determination of softening point
6.	IS 1206 (Part 1) : 1978	Determination of viscosity: Part 1 Industrial viscosity
7.	IS 1206 (Part 2) : 1978	Determination of viscosity: Part 2 Absolute viscosity
8.	IS 1206 (Part 3) : 1978	Determination of viscosity: Part 3 Kinematic viscosity
9.	IS 1207 : 1978	Determination of equiviscous temperature (EVT)
10.	IS 1208 : 1978	Determination of ductility
11.	IS 1209 : 1978	Determination of flash point and fire point
12.	IS 1210 : 1978	Float test
13.	IS 1211 : 1978	Determination of water content dean and stark method
14.	IS 1212 : 1978	Determination of loss on heating
15.	IS 1213 : 1978	Distillation test
16.	IS 1214 : 1978	Determination of matter insoluble in benzene ( <i>WITHDRAWN</i> due to toxic nature of benzene)
17.	IS 1215 : 1978	Determination of matter insoluble in toluene
18.	IS 1216 : 1978	Determination of solubility in carbon disulphide trichloroethylene
19.	IS 1217 : 1978	Determination of mineral matter ash
20.	IS 1218 : 1978	Determination of phenols
21.	IS 1219 : 1978	Determination of naphthalene
22.	IS 1220 : 1978	Determination of volatile matter content

(Continued on third cover)

## *Indian Standard*

# METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS — SAMPLING OF BITUMINOUS MATERIALS

( *Second Revision* )

## 1 SCOPE

This standard covers methods of sampling bitumen and bituminous materials, binders, emulsions, reclaimed asphalt material in their liquid, semi-solid or solid states supplied in any of the following forms:

- a) Cans or small containers, drums, barrels and bags of different sizes depending on the state of the material;
- b) Solid materials in loose form;
- c) Rail tank wagon and tanker trucks or lorries; and
- d) Bulk storage tanks and other types of bulk storage.

## 2 TERMINOLOGY

For the purpose of this standard the following definitions shall apply.

**2.1 Lot** — The quantity of material of the same composition offered for inspection at one time. A lot may consist of the whole or a part of the indented quantity.

### 2.1.1 Formation of Lots

- a) The lot shall be in place before sampling is undertaken.
- b) The basic principle in declaring a lot is that the material in the lot should be as homogeneous as possible with regard to the quality characteristics specified.
- c) Care shall be taken so that the material in a single lot comes from the same source or batch of manufacturer/supplier and to the extent possible from raw material of identical origin.

**2.2 Upper Level Sample** — A sample of upper level is the one taken at a level of one-sixth of the depth of the material below the top surface and in the centre of the container.

**2.3 Middle Level Sample** — A middle level sample is the one taken at a level of one half of the depth of the material below the top surface in the centre of the container.

**2.4 Lower Level Sample** — A sample of lower level is the one taken at a level of five-sixths of the depth of material below the top surface in the centre of the container.

### NOTES

**1** In the case of horizontal cylindrical tanks and tanks of irregular shape, 'upper', 'middle' and 'lower' samples taken at levels corresponding to one-sixth, one-half and five-sixths of the depth of product in the tank are not representative. In such circumstances, samples shall be taken at levels corresponding to one-sixth, one-half, and five-sixths of that volume.

**2** In the case of cone-roof tanks, the sample may be drawn from the centre as near as possible.

**2.5 Average Sample** — A true average or representative sample is such that its composition would be the same as that of any part of the quantity sampled if the whole were mixed to ensure homogeneity. When the contents of the vessel are substantially homogeneous as shown by preliminary examination of upper, lower and middle samples and the cross section of the vessel is uniform, an average sample is usually made up by combining equal parts of samples drawn from levels at one-sixth, one-half and five-sixths of the depth of the liquid below the top surface.

**2.6 Composite Sample** — A composite sample is one taken by combining representative samples of a product from a number of containers that is, from packages in proportion to the contents of each of the containers sampled.

## 3 SAMPLING APPARATUS

**3.1 Bitumen Sampler** — A strong metal vessel of sufficient capacity, the handle of which shall be attached by means not adversely affected by hot bitumen (see Fig.1).

**3.2 Sampling Thief or Tube Method** — The sampling thief or tube shall be either of glass or metal, to reach to within 3 mm of the bottom of the vessel and having a capacity of approximately half or one litre. A metal tube suitable for sampling 250 litre drums is illustrated in Fig. 2. The rings shall be soldered to opposite sides of the tube at the upper end for convenience in holding it.



FIG. 1 BITUMEN SAMPLER

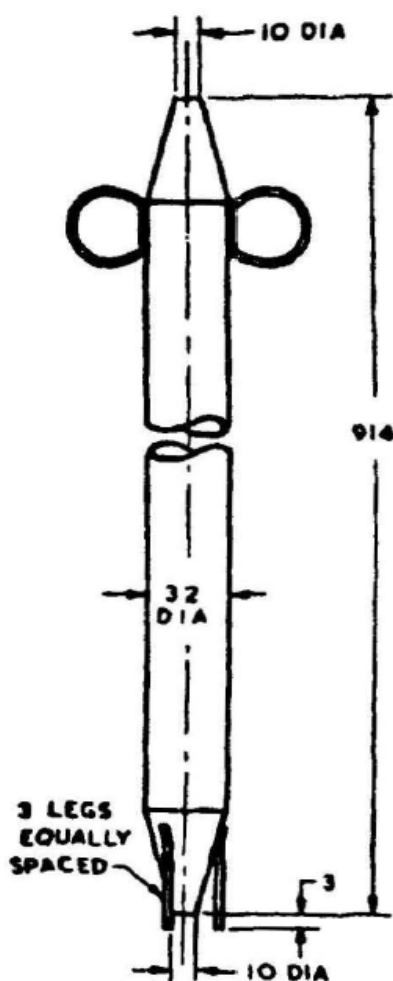


FIG. 2 SAMPLING TUBE

**3.3 Sampling Scoop** — of such dimensions as to enable a core of convenient size to be taken throughout the depth of the package (*see* Fig. 3).

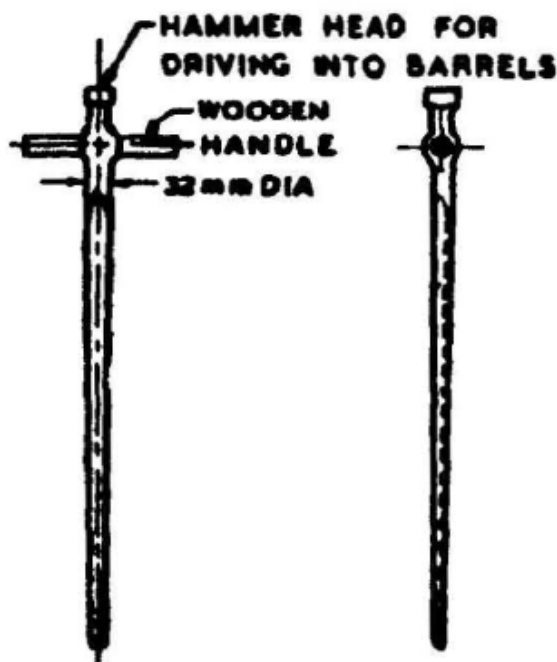


FIG. 3 SAMPLING SCOOP

**3.4 Knife or Spatula** — Provided with a strong, broad steel blade for use with materials which are soft.

**3.5 Hammer and Chisel** — Of any convenient size for use with samples which are hard enough to shatter.

**3.6 Bottle Sampler** — Made of metal with a handle and stopper (*see* Fig. 4).

### 3.7 Sample Containers

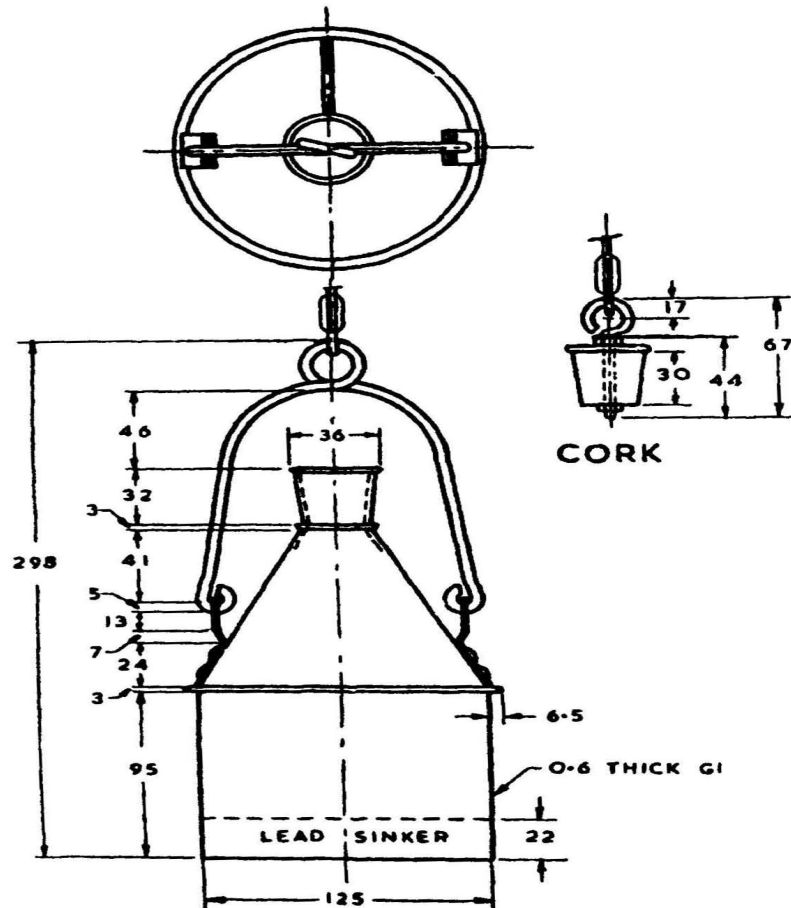
**3.7.1** Containers for the samples of liquid bituminous materials (*see* Fig. 5) shall be small-mouth cans with cork-lined screw caps, except for emulsions. In which case they shall be wide-mouth glass jars or bottles. Containers for the samples of semi-solid and solid materials shall be friction-top cans.

**3.7.2** The size of the sample container used shall be such that it is nearly filled by the sample taken just enough free space such as 5 to 10 percent of the capacity of the container being left to allow for expansion and to enable the contents to be mixed by shaking and not allowing ageing.

NOTE — Sample container should be clean, dry and oil free to prevent sample contamination.

## 4 SAMPLE SIZE

**4.1** The sizes of samples to be taken for testing shall be as follows:



NOTE — A tolerance of 10 percent is allowable on dimensions in millimetres.

FIG. 4 BOTTLE SAMPLER FOR TANK CARS



FIG. 5 (A) SAMPLE CONTAINERS FOR BITUMEN BINDERS



FIG. 5 (B) SAMPLE CONTAINERS FOR BITUMEN

**4.1.1 Liquid Materials**

- a) From small containers, cans, drums and barrels — One litre for each sample.
- b) From bulk storage tanks and drums — 5 litres.

**4.1.2 Semi-solid or Solid Materials**

- a) From barrels; drums; cakes and powdered materials in bags — 1 to 2 kg for each sample.
- b) From bulk — A minimum of 4.5 kg composite or average sample.

**5 PROCEDURE AND SCALE OF SAMPLING****5.1 From Cans or Small Containers, Barrels, Drums and Bags**

**5.1.1 Liquid Materials** — The number of packages to be selected for sampling from the lot shall be in accordance with col 1 and 2 of Table 1. The sample packages shall be selected at random.

**Table 1 Scale of Sampling**  
( Clauses 5.1.1 and 5.1.2 )

Number of Containers in the Lot (1)	Number of Containers to be Selected for Sampling (2)
1	1
2 to 15	2
16 to 50	3
51 to 150	5
151 to 500	8
501 and above	13

**5.1.1.1 Composite sample** — From each of the packages selected for sampling a small quantity of material shall be withdrawn with the help of a sampling apparatus in accordance with the procedure given in 5.1.1.2. The quantity so withdrawn from each of the packages shall be fully representative and proportional to its contents. The material withdrawn from all the packages shall be thoroughly mixed together and then the requisite quantity for testing shall be withdrawn. This shall be the composite sample. An identical composite sample if required may also be made keeping as reference sample under the joint seals of the purchaser and the seller. If the composite sample passes in respect of all the requirements of the specification the lot may be considered to be in conformity to the requirements of the specification.

**5.1.1.2** A sampling apparatus of convenient size shall be lowered into the package so that it touches the bottom, and if the container contains free water its contents shall be rejected and the apparatus is again lowered into the package to such a depth as to avoid the water. The apparatus shall then be withdrawn as before and its contents are used to rinse the sample container and then discarded. The apparatus shall next be lowered slowly as far as possible into the contents of the drum or can, without touching the water layer, if present. The apparatus shall then be closed and withdrawn and the sample allowed to flow gently and without splashing into a sample container.

**5.1.2 Semisolid or Solid Materials** — The number of packages to be selected for sampling from the lot shall be in accordance with col 1 and 2 of Table 1. The sample packages shall be selected at random.



**5.1.2.1** From each of the sample packages from the lot appropriate quantity shall be taken. For this purpose an appropriate sampling apparatus, aid or device may be used which may involve use of hammer, chisel, knife or spatula. If the material is held in small containers with a bung-hole too small to introduce the thief, the contents of the container shall be mixed thoroughly by shaking and the requisite quantity of sample shall be poured out. Care shall be taken to ensure that from each container the material taken is fully representative and proportional to its contents. The material so taken from each of the sample containers shall be thoroughly mixed together or melted together if necessary to form a combined mass of material from which a suitable sample for testing shall be taken. This shall be called the composite sample. If required a similar composite sample may be taken for purpose of preservation for reference under the joint seals of purchaser and the seller. The composite sample shall be required to pass all the specified requirements in order to declare a lot to be in conformity to the requirements of this specification.

## **5.2 From Rail Tank Wagon and Road Tank Trucks or Lorries**

**5.2.1** A careful observation of the condition of the material in regard to the presence of foam, sediment, or free water on top or at the bottom of the material in the wagon/truck shall be made immediately before sampling, and, if such extraneous materials are present in measurable quantities, their volume shall be estimated and samples of each shall be taken and properly labelled. If possible, the foreign materials shall be removed and discarded before sampling.

**5.2.2** Liquid bituminous materials shall, if practicable, be sampled before heating, but if heating is necessary, at no time shall the temperature exceed that required for satisfactory sampling. Semisolid or solid bituminous materials shall be rendered fluid by heating.

**5.2.3** When possible, thoroughly mix the material to be sampled by circulating for several hours before samples are taken. Collect the sample from the draw-off cock at the bottom of the tank in the bitumen sampler. When it is not possible to mix the contents of the tank, or when it is desired to ascertain if the contents of the tank are uniform, take samples by means of a bottle sampler by the method described in **5.3.1**. Close the sample container immediately.

**5.2.4** From each tank wagon/truck the requisite quantity of material (1 lit from a 20 MT tanker) shall be taken to constitute the composite sample for testing, for example, 5 numbers of samples from 25 MT bulk tank. If the composite sample is found to pass all the tests, the material in the tank wagon/truck shall be considered to be in conformity to the requirement of the specification.

If required an identical composite sample may be taken for reference.

## **5.3 From Bulk Storage**

**5.3.1 Liquid Materials and Materials Made Liquid by Heating** — The inlet and outlet to the storage tank shall be closed and a 5-litre sample drawn from the upper, middle and bottom contents. The sample may be taken from drain cocks on the side of the tank if such are available, with the help of a sampler illustrated in Fig.1 and when so taken, enough material shall be allowed to flow through the drain cocks and discarded to ensure a representative sample from the contents of the tank. Otherwise samples may be taken by lowering into the material a suitable bottle sampler, a satisfactory type of which is illustrated in Fig.5. The bottle or can shall be fitted with a stopper which shall be removed by a string or wire attached to it after it has been lowered to the proper depth. The 1 to 5 000 kg lot samples from bulk storage may be tested separately for consistency in order to detect stratification. They may then be combined, thoroughly, mixed, and a 5-litre sample taken therefrom for other tests that may be required for determining average characteristics of the material.

**5.3.2 Solid Materials in Loose Form** — Mix the piece representing the total quantity on a clean hard surface and proceed as follows:

Place the broken down samples on a clean impervious surface, mix by shoveling and heap into a cone by depositing separate small quantities one on top of the other, the cone being formed symmetrically throughout, with its apex always in the same vertical line.

Alternatively, shovel the material into a funnel having a sort cylindrical stem about 51 mm in diameter. Support the funnel in a vertical position above the top of the cone, and raise it (without lateral displacement) as the cone grows higher to allow the material to flow, out gently and not fall from a height. Form a new cone twice. In a similar way, flatten the third cone by pressing it with a metal sheet or any other suitable appliance, the flattening being carried out symmetrically, thus giving a mass of approximately uniform thickness and diameter. Divide the sample at this stage in quarters by pressing through it a metal cross constructed from four arms or blades which meet at a common centre, held at right angles to each other by stays. Remove a pair of opposite quarters completely and reject them. Mix the two remaining quarters together thoroughly, reduce in bulk by further quartering, and repeat the procedure until a final sample of suitable quantity is left. The gross sample shall not be less than 25 kg from which shall be selected 1 to 1.5 kg of the composite sample for tests. If the composite sample satisfies all the specified tests, the material in the lot shall be declared to be in conformity to the specified requirements.

## 6 PRECAUTIONS

**6.1** In addition to the general precautions given in **6.2**, in sampling for certain tests, some special precautions need to be observed. Such precautions are prescribed in the relevant standards for method of tests.

**6.2** A sample shall not include material other than that to be sampled and shall not become altered, for example by evaporation of volatile constituents or by oxidation, in the process of sampling. The following precautions shall be observed in sampling:

- a) Official samples shall be taken by, or under the immediate supervision of a person of judgment, skill and experience in sampling.
- b) Sampling of bituminous material is normally associated with the risk of heat burns due to spilling and splashing of the hot bitumen. It is necessary to wear appropriate personal protective equipment while collecting the hot samples.
- c) The surrounding area should be adequately illuminated, should have safe and easy access for collection of the samples.
- d) Presence of hydrogen sulphide in sampling area should be tested before starting the sampling process.
- e) The sampling apparatus, including cords or other ancillary gear and sample containers shall be dry and free from any substance which may contaminate the sample.
- f) The sampling apparatus shall be filled and allowed to drain at least twice before drawing the sample and the sample container shall be rinsed at least twice with the material obtained during the third or any subsequent filling of the sampling apparatus and shall be allowed to drain before being used to contain the actual sample.
- g) During sampling operations, the material being sampled shall be protected, as far as possible, from the effects of wind and weather. The sample containers shall be closed immediately after the sample has been taken.
- h) Sampling should be done through a platform and the person carrying out the sampling should be harnessed securely to the platform and not on the truck. Personal Protective Equipment shall be worn during sampling. Full body overall with fire resistant material, face shield, eye goggles, safety helmet, heat resistant gloves, H<sub>2</sub>S detector, Safety Shoes (heat and splash resistant) shall be worn by the person carrying out sampling.

Facilities for safety shower and eye wash are to be made available in the immediate vicinity of the sampling area.

- j) Liquid materials in tanks and other bulk containers shall be sampled by the appropriate method

prescribed in this standard. The sample shall be drawn through dip hatches, manholes or other opening giving direct and unconfined access to the bulk of liquid. Samples shall not be drawn from dip-pipes or other fittings. Gauge glasses or drain fittings shall not to be used for sampling unless specified.

- k) Care shall be taken to prevent contamination of sample by water or any other foreign material. This may also cause unsafe environment of work.
- m) If there is any reason to suspect that a sample or a set of samples may not be fully representative, a further sample or set of samples shall be taken. The necessity for taking a second or 'check' sample, or set of samples, generally arises due to failure or faulty labelling.
- n) All metal sampling gear should be made of non-spark generating materials.
- p) Rubber stoppers or corks shall not be used for closing vessels containing samples.
- q) Care shall be taken to prevent contamination of the stock, for example, by dirt or other extraneous matter.
- r) It is advisable to take more than one set of samples for check purposes in case of dispute, leakage or breakage in transit or for any other reasons.
- s) Every possible precaution against fire hazard shall be taken when sampling flammable materials like cutback bitumen, etc.

NOTE — The person involved in sampling should use adequate Personal Protective Equipment as per the Material Safety Data Sheets (MSDSs) of the material to be sampled.

## 7 LABELLING, PACKING AND STORAGE OF SAMPLES AND SAMPLE CONTAINERS

### 7.1 Labelling

Sample containers shall be labelled. Gummed labels may be used on glass surfaces but in general wired-on labels shall be preferred. Labels shall be permanently marked. The following information shall be recorded on the label:

- a) Sample reference number;
- b) Place at which sample was drawn;
- c) Date of sampling;
- d) Initials or other identification mark of sampling officer;
- e) Grade of material;
- f) Quantity represented by sample;
- g) Tank number, package number and type, name of the point of dispatch;
- h) Type of container from which sample was drawn and type of sample (upper/middle/average etc); and
- j) The name of the manufacturer/supplier.



## **7.2 Packing**

Sample containers shall be sealed properly to prevent tampering. The type of packing used for samples which are to be transported depends largely on the means of conveyance. Wooden cases or strong cardboard cartons are recommended for cans containing samples. The space round the sample container shall be filled with material, such as saw dust; when such materials are

used, the cork or stopper shall be covered with a paper or viscose cap in order to prevent contamination of the sample on opening.

## **7.3 Storage**

Samples of materials which may be affected by light or heat shall be stored in a cool, dark place, periodical examination shall be made for leakage.

## ANNEX A

( Foreword )

## COMMITTEE COMPOSITION

Bitumen, Tar and Related Products Sectional Committee, PCD 06

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CSIR-Central Road Research Institute, New Delhi	PROF SATISH CHANDRA ( <b>Chairman</b> )
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*Member Secretary*

SHRIMATI D. UMA  
SCIENTIST 'D' (PCD), BIS



*(Continued from second cover)*

However, the Committee responsible for the formulation of standards in the field of bitumen, tar and related products decided to publish these Indian standards separately for each test so as to make it user friendly. Accordingly, second revision of the standard, IS 1201 'Methods for testing tar and bituminous materials — Sampling' has been taken up to formulate individual standard.

In this revision, the following major modifications have been made:

- a) Scope has been extended for Bituminous binders and emulsions;
- b) The term 'Top Sample' has been renamed as 'Upper Level Sample (2.2)';
- c) Terms 'Pipe line sample' and 'All level sample' have been removed; and
- d) Precautions (6) has been modified;

Sampling involves the act, process and technique of selecting a representative part or portion of a bulk or lot or mass for the purpose of determining parameters or characteristics of the whole. Samples are taken for the purpose of visual assertion and laboratory examination or for preservation for record. The examination may be made to determine

- a) the characteristic quality of the material in the lot,
- b) the extent of variation in quality in the different portions of the material, and
- c) conformity to the specifications/specified requirements.

The Composition of the Committee responsible for formulation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

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### Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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